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AN INVESTIGATION OF EXPERIMENTAL TECHNIQUES FOR OBTAINING PARTICULATE BEHAVIOR IN METALLIZED SOLID PROPELLANT COMBUSTION*

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ABSTRACT

A continuing investigation is being conducted to obtain quantitative data that can be used to relate propellant composition and operating environment to the behavior of solid particulate within the grain port and exhaust nozzle. The techniques employed are monochromatic high-speed motion pictures of strand burners and slab burners in a cross-flow environment, SEM analysis of post-fire residue (strand, slab, and motor), determination of D_{32} across the exhaust nozzle using measurements of scattered laser light, holograms of burning propellant strands and slabs in a cross-flow environment, and automated retrieval of particle data from holograms. Progress and data examples for each technique are presented.

INTRODUCTION

At the Naval Postgraduate School (NPS), a continuing investigation is being conducted to obtain quantitative data that can be used to relate propellant composition and operating environment to the behavior of solid particulate (Al, Al_2O_3) within the grain port and exhaust nozzle.

These data are needed in order to (1) improve solid propellant performance predictive capabilities, (2) provide needed input related to AP-aluminum interactions for current steady-state combustion models, and (3) provide in-motor particle size distributions which will allow more accurate predictions of damping in stability analyses. The techniques employed are high speed motion pictures of strand burners and slab burners in a cross-flow environment, SEM analysis of post-fire residue (strand, slab, and motor), determination of D_{32} across the exhaust nozzle using measurements of scattered laser light, and holograms of burning propellant strands and slabs in a cross-flow environment. In addition, work has been initiated to develop automatic data retrieval methods for obtaining particle size distributions from holograms taken of the combustion of solid propellants. Once developed, these techniques/diagnostic methods could be readily employed for obtaining the needed data discussed above from a series of tests in which propellant composition, motor geometry, and operating environment are systematically varied.

In FY'82 the motion picture and holographic techniques were successfully demonstrated using propellant strands burned at operating pressures of 34 and 68 atm. and with up to 15% aluminum. Fourteen μm resolution was obtained in the high speed motion pictures with a 1.12x magnification (and very small depth of field) and an eleven μm resolution was obtained in the holograms. In addition, initial measurements of D_{32} were made using measurement of scattered laser light at the exhaust of a small rocket motor. Several needed improvements to the diagnostic technique were made in FY'83. These were:

(1) Use of laser light illumination and narrow pass filters with the high speed motion pictures in order to eliminate the flame envelopes around the burning particles.

(2) Use of photo-diode linear arrays in place of a translating photo-diode in order to improve the accuracy of the measurement of scattered laser light.

(3) Incorporation of a second diode array at the nozzle entrance so that particulate changes across the nozzle could be measured.

(4) Increasing the power density that reaches the holographic plate by both laser and two-dimensional motor design changes.

(5) Initiation of the development of a system for the automatic retrieval of particulate data from holograms.

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HIGH SPEED MOTION PICTURES

In FY'83 the high speed motion picture investigation of burning propellant strands has been continued using a combination of monochromatic and white light illumination. The current illumination method uses three illumination sources during one test (Fig. 1); 2,500 watt white light rear illumination, 1,200 watt white light side/front illumination, and 0.8 watt side/front illumination at 488 nm. With this illumination method a rotating filter disc is placed between the combustion bomb window and the camera lens. This provides alternating frames with high intensity white light illumination and filtered 488 nm illumination. Initial tests with this technique have resulted in films in which the particle flame envelopes are eliminated as desired. Currently, work is being directed at optimization of f-stop, magnification, filming rate, and propellant thickness in order to obtain the best film exposure.

The obtainable results using (1) normal white-light front illumination, (2) intense back-lighting with white light, and (3) monochromatic illumination will be shown in the oral presentation.

DETERMINATION OF MEAN (D_{32}) PARTICLE SIZE USING MEASUREMENTS OF SCATTERED LASER LIGHT

In FY'82 and FY'83 a dual beam apparatus was developed which simultaneously measured particle size (D_{32}) at the entrance and exit of an exhaust nozzle of a small solid propellant rocket motor (Figs. 2 and 3). The diameters were determined using 1024 element linear photodiode arrays to measure diffractively scattered laser light (Fig. 4). He-Ne illumination was used at the nozzle exhaust but significant 632.8 nm radiation within the motor combustion zone required the use of argon laser (488 nm) illumination in that region. The apparatus has been calibrated using spherical glass beads and non-spherical Al_2O_3 suspended in water. The calibrations obtained using the glass beads were in reasonably good agreement with the theoretical profile (Ref. 1-10). The calibration using non-spherical Al_2O_3 was not in as good agreement and the size of the Al_2O_3 powder affected the correlation.

Measurements are made with and without the particles present in order to be able to help eliminate the scattering caused by the optics and motor hardware. A normalized intensity is then calculated

$$\bar{I} = \frac{I_\theta - I_{\theta_{\text{no particles}}}}{I_{\theta=0} - I_{\theta=0_{\text{no particles}}}} \tag{1}$$

where $I_{\theta=0}$ is obtained by extrapolating the scattered light profile to the optical axis ($\theta=0$) where the dominant light intensity is from transmitted light. The diode array currently measures the scattered light between angles of 0.003 radians (0.17°) and 0.054 radians (3.1°). The first diode would be damaged by the intensity of the transmitted light if it were located on the optical axis.

Either the universal profile or one of the calibration curves can be entered at a selected value of $\bar{I}$ (or θ, at values where the best calibrations occurred) to obtain $\bar{\theta} = \frac{\pi D_{32}\theta}{\lambda}$ and, therefore, D_{32}.

Alternatively, the ratio of $(I_\theta - I_{\theta_{\text{no particles}}})$ obtained at two scattering angles can be used to obtain D_{32}. For example, if a Gaussian approximation is made to the theoretical scattering profile (Ref. 11) then

$$\frac{I_2}{I_1} = \exp\left[-\left(\frac{0.57\pi D_{32}}{\lambda}\right)^2 (\theta_2^2 - \theta_1^2) \right] \tag{2}$$

During the motor firing, the location of the laser beam in the exhaust jet was found to be critical. If directed too far to the rear of the nozzle exit, either it missed the jet entirely due to the deflection of the jet or the particle number density was too low. Both problems resulted in a particle scattering profile that was nearly identical to the zero particle scattering profile. With the current motor the beam was located 8 cm to the rear of the nozzle exit.

The tests conducted are summarized in Table I. The first series of tests were conducted at 53 atm. There were two significant results: (1) there was insufficient light through the motor to get a scattering profile, and (2) the measured D_{32} in the exhaust was large (30-50 µm). Photographs of collected exhaust products showed the apparent reason for this large D_{32} to be in the presence of a small number of large irregularly shaped agglomerates, which were not Al or Al_2O_3 (typically 0.5-5 µm spheres). These agglomerates were most probably inhibitor or binder char. Their influence was significant, as even a very few large particles greatly increase the measured D_{32}. The sensitivity of the technique to irregularly shaped particles had already been demonstrated during the calibration using Al_2O_3 powder. Repeated tests resulted in different D_{32} at the nozzle exhaust, apparently from different sizes of char agglomerates at the time the diode scan was made.

The tests made at approximately 34 atm. (runs 3, 4 and 5, Table I) were made to determine, (1) if the size of the exhaust char agglomerates were sensitive to combustion pressure, and (2) if the lower chamber pressure would permit measurement of scattered light through the motor. In addition, repeated runs were made to acquire the diode data at different times during the run to determine whether the data was significantly time dependent. It was found that data could be obtained through the motor. Comparisons with the universal curve and the small glass bead calibration curve yielded reasonable values for D_{32} of 16 and 12 µm, respectively. It was further determined that D_{32} measured at the nozzle exit was sensitive to burn time, as shown by the increasing values of D_{32} with increased time. Since the spherical Al/Al_2O_3 particles (in the collected exhaust products) were still in the 0.5 to 5 µm size range, the change in measured D_{32} was apparently due to changes in char agglomerate size during the run.

In run 6 an end-burning grain was used in an attempt to reduce or eliminate the large char agglomerates. The results were: (1) a significant reduction in measured D_{32} (however, it was still larger than the Al/Al_2O_3 spheres), and (2) the larger optical path with particles (increased optical density) precluded the measurement of any scattered light through the motor cavity.

Two approaches are currently being used to improve this diagnostic technique; improvement of the propellant and improvement of the instrumentation. Rather than one sweep of the diode arrays during one firing, seven will be accomplished. This is being made possible by expanding the memory of the multiprogrammer and by utilization of the HP9836S computer in place of the HP85. This should result in a more statistically valid data sample. The Air Force Rocket Propulsion Laboratory (AFRPL), is currently fabricating GAP propellants to replace the HTPB propellants which are currently being used. This should result in "cleaner" exhaust products and improve the accuracy of the measured particle sizes.

HOLOGRAPHIC INVESTIGATION OF SOLID PROPELLANT COMBUSTION

The two-dimensional motor was designed for two opposed, end-inhibited propellant slabs. A 8.9 mm diameter, high-quality glass window port was positioned where the laser beam entered the motor. The glass window port through which the laser beam exited the motor was enlarged to 18.5 mm in diameter. This increased the laser power density incident on the photographic plate, facilitating exposures through a more opaque chamber medium. These glass windows were moved as close as possible (given the current motor configuration) to the propellant to minimize the combustion chamber free volume, and to minimize the flow-disturbing effects produced as the window-protecting shutters were retracted.

The windows are centered at the lengthwise midpoint and inboard edge of the largest propellant slab. The smallest propellant slab was out of view of the smaller window port to facilitate maximum viewing of the combustion chamber volume and also to create a reasonable cross-flow environment.

A Plexiglas spacer was mounted on each window shutter block as shown in Figure 5. The spacers were shimmed during motor assembly to insure that both sides of the propellant slabs were in physical contact with Plexiglas. This reduced the possibility of uncontrolled side burning, kept chamber free volume to a minimum, and facilitated a cross-flow environment.

Retractable shutters protected the glass windows prior to laser activation and were retracted just prior to laser firing. The nitrogen purge system was modified (Fig. 6) to direct nitrogen across each window throughout the firing/hologram recording sequence.

The ignition and ignition assembly procedures were also modified to eliminate the need for black powder as an igniter (Fig. 7). A time-delay was activated at ignition initiation. At the expiration of the time delay the spring-loaded shutters were released. The rising shutters in turn tripped a microswitch, which triggered the holocamera shutter and fired the laser simultaneously. A photodiode was added to the laser system to sense laser firing in order to mark the precise time of laser activation on the pressure-time plot.

The propellant slabs produced insufficient pressure to pressurize the combustion chamber to the desired 34 atm. The chamber mounted above the combustion chamber (Fig. 7) was pressurized with nitrogen and the burning propellant slabs provided the remaining pressure. In the opposed slab configuration, the propellant provided approximately fourteen percent of the desired steady state pressure. The majority of the chamber pressurization was provided by the nitrogen introduced downstream of the propellant. Use of the upper chamber reduced the tendency of the pressurizing nitrogen to recirculate into the combustion region.

A 0-80 screw was mounted outside the large exit window in the scene beam path and was used to provide a scale for particulate sizing in the reconstructed hologram.

The propellant slabs were rough-cut, then hand rubbed to the final dimensions (13 X 29 X 1 mm) to remove loose material which would cause the inhibitor to debond during firing. The slab was epoxy bonded to the propellant support base and inhibited with a thin coating of General Electric Hi-Temp Gasket (Red RTV), then allowed to cure for twenty-four hours. The propellants were provided by the Aerojet Solid Propulsion Company and are as described in Table II.

TABLE II

Propellant Composition and Metal Additive Particle Size

Propellant Designation	Binder % Weight	Oxidizer % Weight	Metal % Weight	Mean Metal Diameter, microns
WGS-5A	HTPB 12	AP 83	AL 5	75-88
WGS-6A	HTPB 12	AP 83	AL 5	45-62
WGS-7A	HTPB 12	AP 83	AL 5	23-27
WGS-7	HTPB 12	AP 83	AL 5	6-7
WGS-9	HTPB 12	AP 78	AL 10	23-27
WGS-10	HTPB 12	AP 73	AL 15	23-27
WGS-ZrC	HTPB 14	AP 84	ZrC 2	23, irregularly shaped
WGS-G	HTPB 14	AP 84	G 2	50X20X7, flakes

The present investigation resulted in the development of a technique for obtaining good quality holograms of burning solid propellant in a two-dimensional motor with smokeless metal additives and with aluminum additives which used powder sizes greater than approximately 23 microns and additive mass contents of 5 percent or less by weight.

The techniques developed provided a greater range of data and more consistent runs than were possible with the original apparatus. However, shutter retraction noticeably disturbed the flow as evidenced by (1) the location of many of the particulates in the reconstructed image nearer the large exit window and not between the burning propellant slabs as would be expected under steady-state, cross-flow conditions, and (2) a small pressure rise following shutter retraction.

Very recently the propellant bonding procedure has been changed. The two propellant slabs (1, 2, or 3 mm thick) are bonded between two pieces of glass similar to the method used by Gany and Caveny (Refs. 12 and 13). This has eliminated the need for window-protecting shutters and provide a more realistic flowfield for the combustion products. The glass remains clear during the burn, allowing a large viewing field. This procedure has resulted in very good quality holograms of the 2-D motor combustion process. Slides taken from various holograms will be shown in the oral presentation.

AUTOMATED RETRIEVAL OF PARTICULATE SIZE DATA FROM RECONSTRUCTED HOLOGRAMS

Automatic retrieval of particulate diameter data from reconstructed holograms is necessary if meaningful amounts of needed data are to realized in a reasonable time period. Various techniques have been suggested that range from complete digitization to man-in-the-loop optical methods. A reasonable near-term solution appears to be a combination of both optical and digital methods. BRL (Ref. 14) has provided a limited demonstration of one such method, based upon the Quantimet 720 Image Analyzer.

The end goal of the NPS effort is to be able to obtain particulate diameter data from a given hologram using a reasonable amount of man-hours/computer time. Some form of image processing with a man-in-the-loop may provide a more realistic method than completely automated methods.

The NPS effort is based upon the Quantmet 720, used in conjunction with holographic reconstruction with a 1-watt krypton laser. The Quantimet 720 produced by Cambridge Instruments Inc. is a general purpose television type image analyzer that is capable of recognizing and measuring particles by distinguishing differences in gray levels. The effort was initiated in mid-FY'83. The initial work has been directed at obtaining particle size data from a photograph taken at one location within a reconstructed hologram. Subsequent work will be directed at speckle suppression methods and obtaining data directly from traverses through the reconstructed hologram.

Two dimensional rocket engines are fired through an apparatus that triggers a Q-switched, pulsed ruby laser at any desired time of the burn cycle to produce a hologram of the combustion products. The holographic scene beam is passed through a diffuser to reduce the schlieren effects produced by the flame envelopes that surround the numerous burning particles. Unfortunately, the use of a diffuse scene beam causes speckle which is recorded in the hologram. To provide a reference for sizing the particles, a 0-80 screw is placed outside the burning motor enclosure but within the depth of field of the hologram. Currently, photographs are taken of the screw and the areas of interest within the combustion zone. Prints and transparancies have been produced, however it has been found that the prints provide a better image for analysis on the Quantimet. The actual scene area under investigation is studied to obtain a particle size distribution. Figure 8 is a photograph of a reconstructed hologram of propellant WGS-G (Table II) taken at 36 atm. Table III is the particle size distribution obtained from a portion of Figure 8.

TABLE III

Particle Size Distribution from Fig. 8

d(μm)	Number of Particles
0-70	11
71-100	6
101-123	5
124-142	2
143-159	2
160-226	1
>226	1

Several observations can be made from Fig. 8. The large agglomerates are binder or inhibitor char, but the image analyzer counts these large objects as particles. Manual editing of the hologram before particle counting can simplify the particle sizing/counting process. Also evident is a non-uniform background illumination caused by smoke and, possibly, non-uniform laser illumination. The latter can be examined using holograms of non-burning environments. Finally, the speckle is seen to reduce the hologram quality and limit the size of discernible particles.

Currently, the largest source of error and the most difficult problem to overcome is speckle. Speckle has two effects on the reconstructed image. It produces black images in the background which cannot be readily distinguished from real particles, except perhaps by size and/or color gradients. The second observed effect of the speckle is that it gives the actual particle a "swiss cheese" like appearance, which alters the area and perimeter of the particle. The Quantimet ignores the holes within the particle when measuring area, but significant error is introduced when the perimeter is altered. The first effect limits the size of particles that can be measured.

In areas of low illumination it is also difficult to distinguished when one particle is darker than another, as well as distinguishing where a particle ends and background or speckle begins. Presently, the skill and methodology of the operator are key factors in determining the size of particles.

Another source of error in the image analysis results from unequal illumination in the hologram. Two particles can be in focus, yet one can be darker than the other due to the amount of illumination on it. This leads to errors in measurements since the Quantimet is programmed to recognize from gray level 0 (which is black) to a set gray level, i.e., it recognizes all particles darker than a set gray level. The eye tends to smooth out small differences in gray levels so that particles appear to be of a uniform gray level or shade. However, the Quantimet recognized these small differences in gray levels and views a particle as a number of concentric circles, each of a different gray level. The darkest level is in the center and decreasing values occur with increasing radial distance from the center. If a set gray level is employed, two equal sized particles photographed at different illumintions will display different areas. The particle that has received less illumintion will generate a greater area.

Future plans are primarily aimed at suppressing speckle since it introduces the greatest error and limits the size of particles which can be measured. As stated earlier, speckle can be suppressed by spatial averaging techniques. A computer controller, PDP-11/04, has been purchased for the Quantimet 720 which allows a number of frames to be stored in internal memory or on a mass storage device. Using software provided by Quantimet, a program can be written which will take a number of frames, sum them together and display an image which is an average of the frames. By varying a parameter, in each frame, such as aperture or illumination angle, an image can be constructed which is the spatial average of a number of frames. This procedure may suppress speckle, which would allow one to measure smaller particles and decrease the effects of speckle on area measurements. In addition, a Quantimet interactive light-pen (image editor) is being purchased which should reduce the time required to analyze the particle data. Future holograms of burning propellant will be made using both diffuse and collimated scene beams. The diffuse scene beam smears the schlieren effects, resulting in better resolution when viewed using the eye. However, the image analyzer may be able to disregard the schlieren effects more readily than the effects created by the speckle.

A motorized XYZ, computer controlled stage has also being purchased. This will allow stepping through a hologram in increments as small as 0.1 μm for determination of when a particle is in focus. The determination of when a particle is in focus will be done with the computer controller and a program that defines a particle as being in focus when the shading that surrounds a particle is less than a certain value. This is possible since particles in focus are characterized by being darker and having almost no shading around the edges. This method will enable particle volume measurements to be obtained. Probable problem areas are determination of the optimum step size and how to handle changing illumination as one moves through the hologram.

SUMMARY

In summary, various techniques are being used to obtain particulate data within the grain port and nozzle of solid propellant rocket motors. Each of the diagnostic techniques (high-speed motion pictures, SEM analysis of collected exhaust, measurement of scattered laser light, holography, data retrieval from holograms) has its unique advantages and disadvantages. If care is taken to optimize each technique, meaningful results can be obtained using all of the approaches investigated to date.

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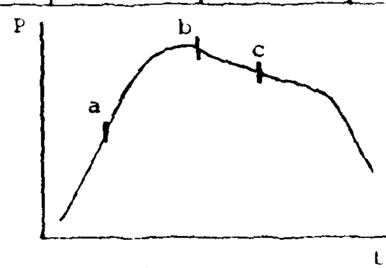
TABLE I. DATA SUMMARY

Propellant contained 2% by weight of Al (40 μm)

Run	1	2	3	4	4a (Nozzle Entrance)	5	6*
Date	15 Sep 82	19 Sep 82	21 Sep 82	23 Sep 82	23 Sep 82	24 Sep 82	25 Sep 82
Maximum Pressure (psia)	775	775	555	565	565	495	455
Readings Taken (psia) (see fig. below)	760 (b)	760 (b)	395 (a)	545 (b)	545 (b)	465 (c)	415 (c)
D_{32} Average, Universal Curve (μm)	52	39	24	37	16	56	16
D_{32} Average Calibration Curve Comparison (μm)**	38 m	30 m	20 s	30 s	12 s	40 ℓ	13 s



(Sample pressure-time trace showing points where data was taken)

*End-burning grain

**s--small particle calib.
m--medium particle calib.
ℓ--large particle calib.

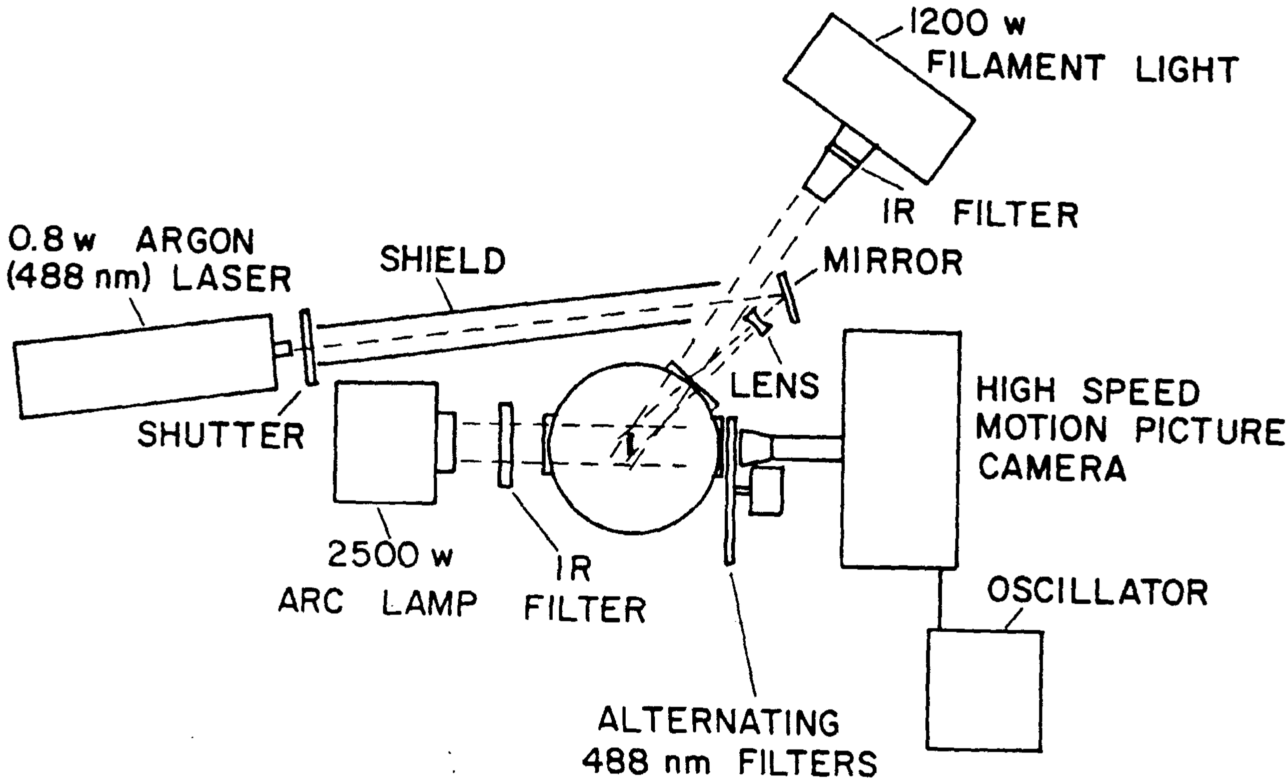


Figure 1. Schematic of Combustion Bomb Apparatus.

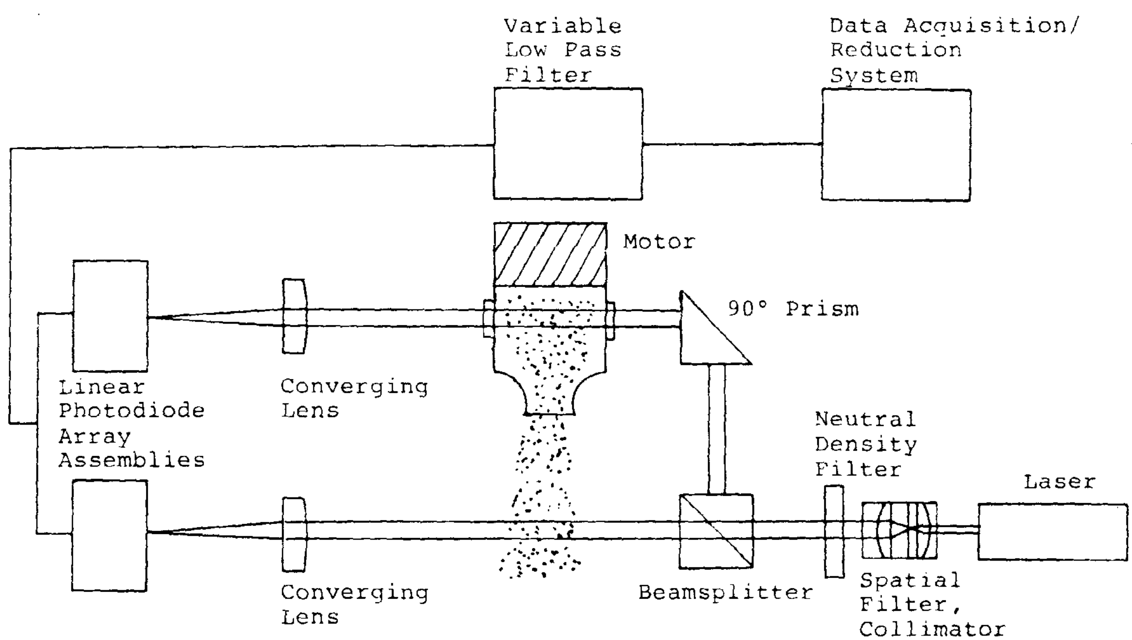


Figure 2: Schematic Diagram of Diffractively Scattered Light Apparatus

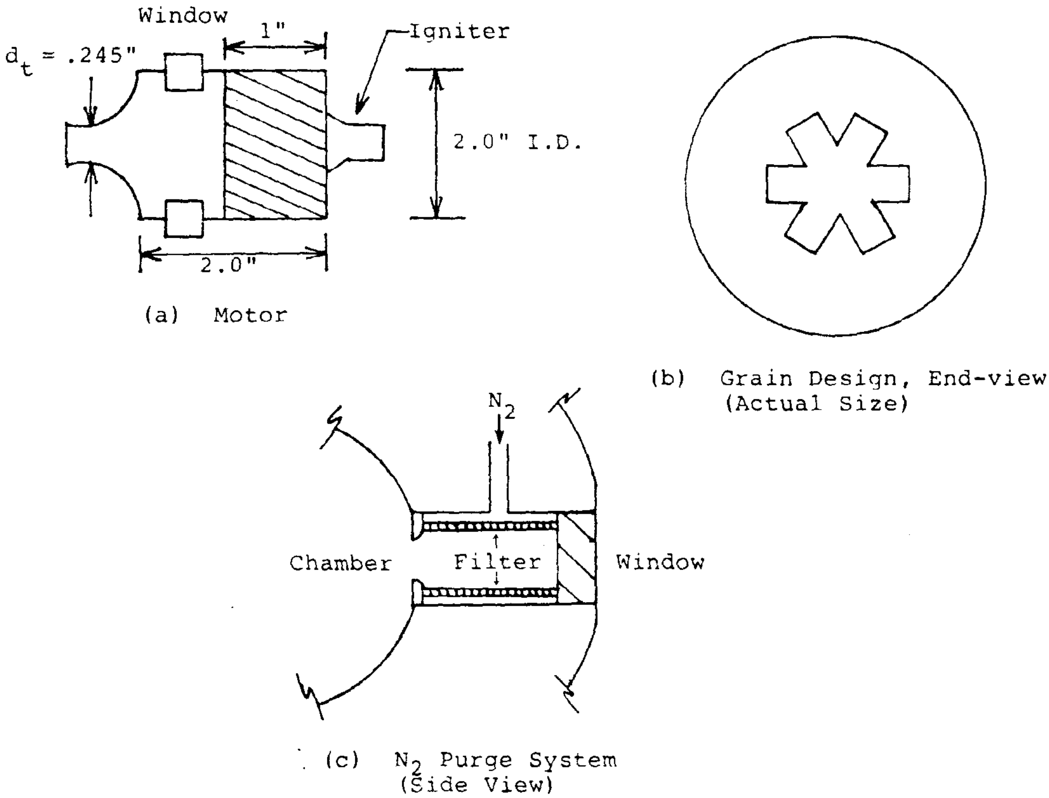


Figure 3 : Motor Components

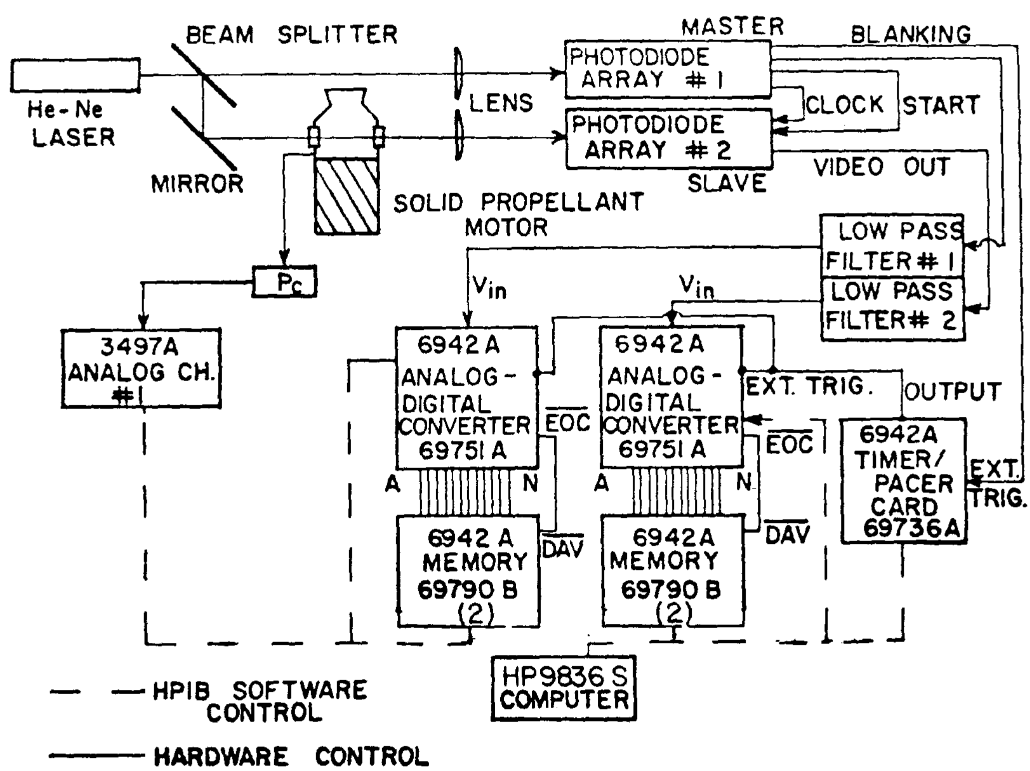


Figure 4 : Schematic of Data Acquisition System

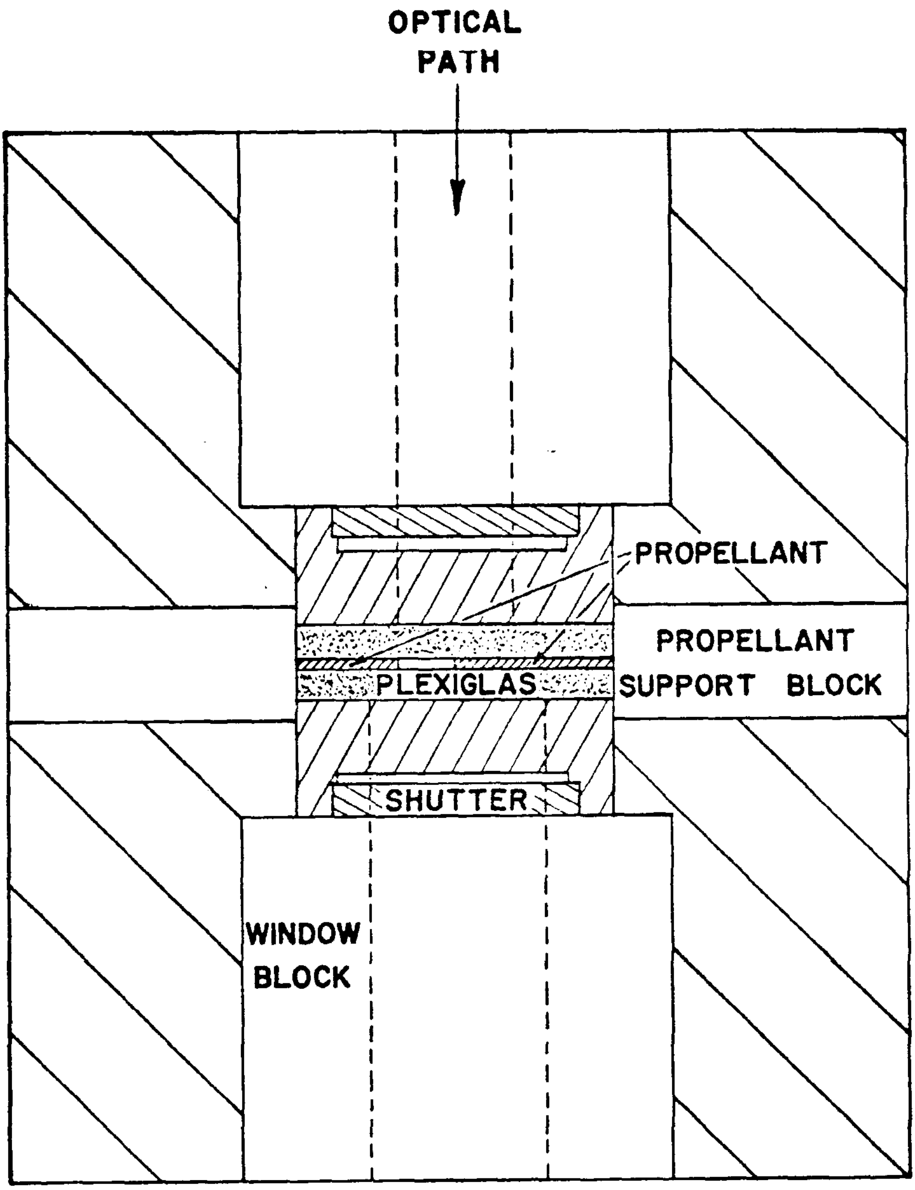


Figure 5. Plexiglas Spacer Arrangement, Top View

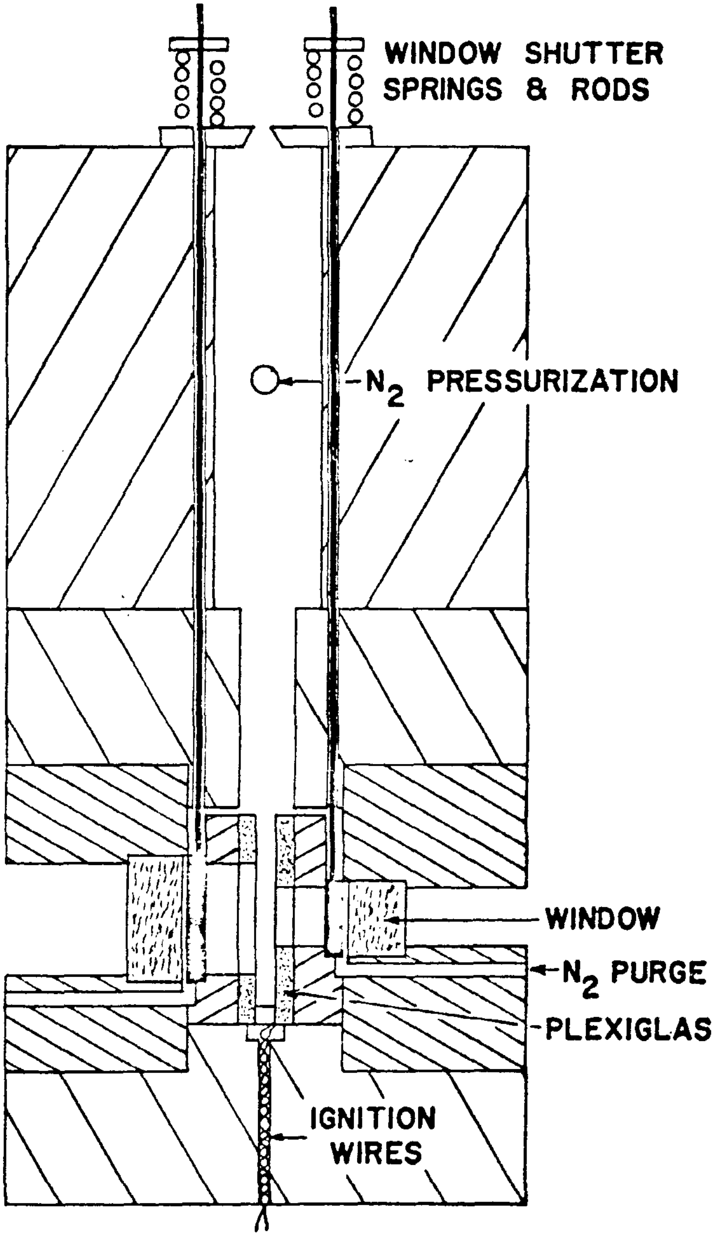


Figure 6 . Motor Line Drawing, Side View

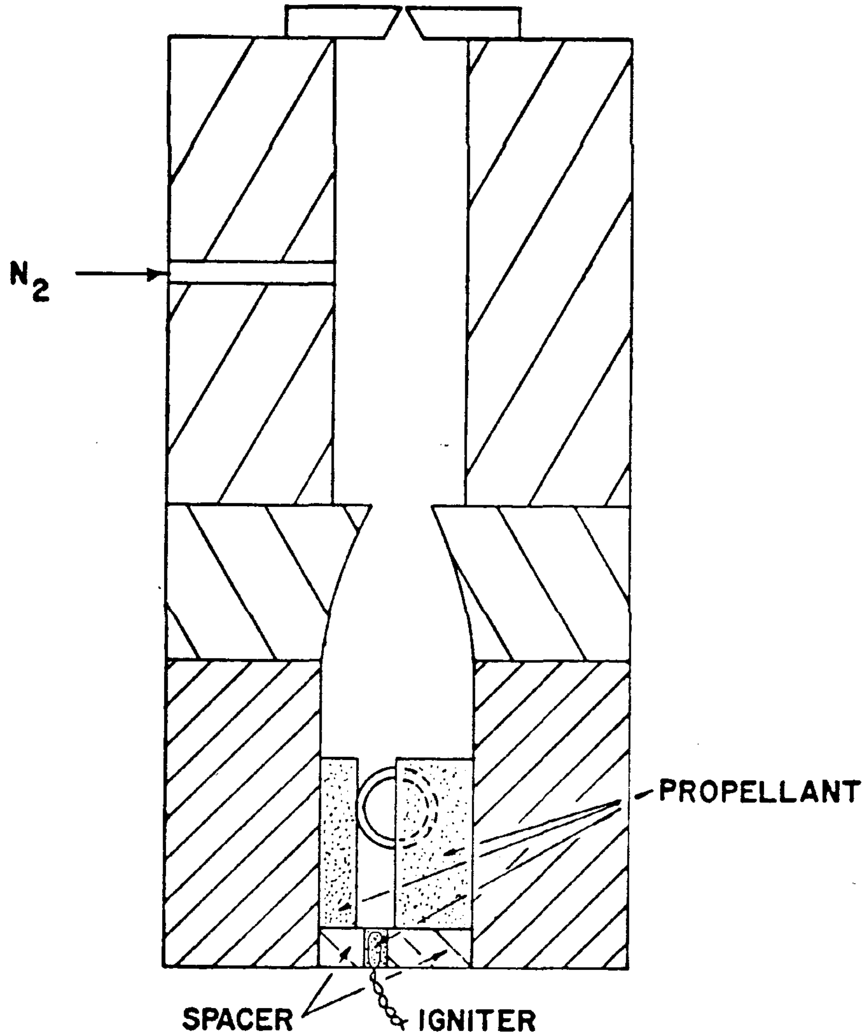


Figure 7. Motor Line Drawing, Window Side View

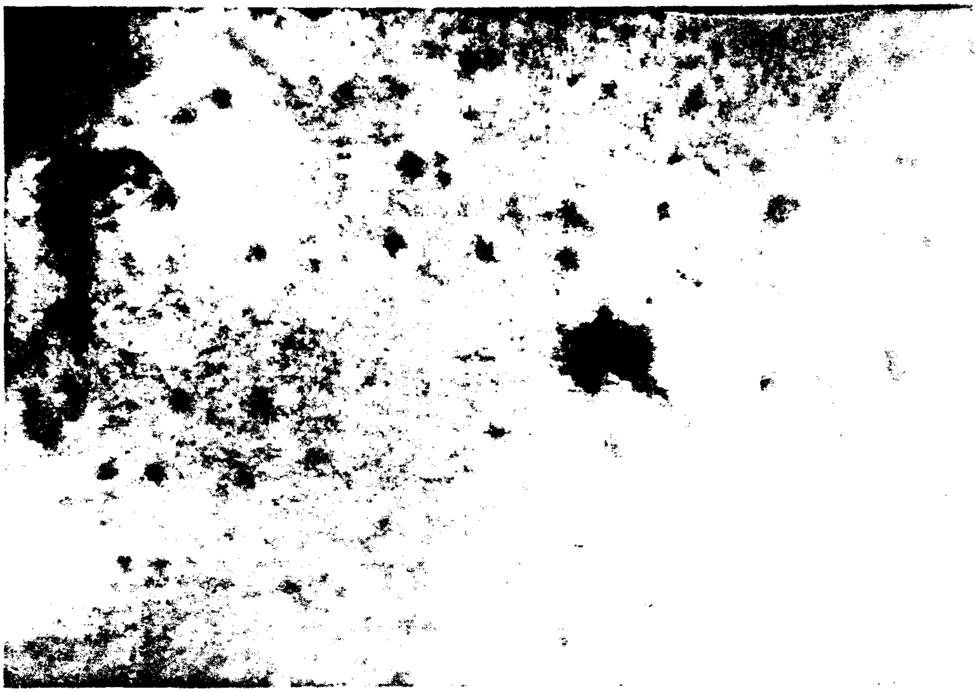


Figure 8. Photograph of Reconstructed Hologram from Propellant Combustion with Stability Additives.